

## Sovereign Consulting Inc.

## OPERATIONAL UNIT 1 (OU1) McCORMICK COUNTY, SOUTH CAROLINA

## **QUALITY ASSURANCE PROJECT PLAN (QAPPS)**

for

TIER 1 - REMEDY SCREENING
[PROOF OF PRINCIPAL (POP) TESTING]

DRAFT FOR REVIEW/APPROVAL

NOVEMBER 8<sup>TH</sup>, 2017

#### **SUBMITTED TO:**

Black & Veatch 1120 Sanctuary Parkway, Suite 200 Alpharetta, GA 30009

#### **ATTACHMENT SAP3**

of the Sampling and Analysis Plan

DV031.001



## Table of Contents

SIGNAT	URE PAGE:	1
1.0 INTE	RODUCTION	2
2.0 PRC	DJECT DESCRIPTION	2
3.0 QUA	ALITY CONTROL ORGANIZATION (SOVEREIGN)	2
4.0 QUA	ALITY ASSURANCE OBJECTIVES	2
5.0 APP	PROACH TO QA/QC	3
5.1 Sa	ample Characterization	3
5.2 W	/aste Rock Testing	4
5.2.1	Initial Qualitative Screening of Waste Rock	4
5.2.2	Utilization Testing (USBM Modified Method)	4
5.2.3	Unsaturated Zone Amendment Screening	4
5.2.4	Transition Zone Amendment Screening	5
5.2.5	Saturated Zone Amendment Screening	5
5.3 Pi	it Lake Tests	5
5.3.1	Pit Lake Floor Encapsulation	5
5.3.2	Pit Lake Neutralization	6
5.4 Da	ata Quality Indicators	6
5.4.1	Precision	6
5.4.2	Comparability	6
5.4.3	Completeness	6
6.0 QA I	FILES	7
7:0 REF	FERENCES	7

Sovereign Consulting Inc.

**SIGNATURE PAGE:** 

# OPERATIONAL UNIT 1 (OU1) McCORMICK COUNTY, SOUTH CAROLINA

## **QUALITY ASSURANCE PROJECT PLAN (QAPPS)**

for

## TIER 1 - REMEDY SCREENING [PROOF OF PRINCIPAL (POP) TESTING]

Print Name:	Date:	· 
Black & Veatch		
Print Name:	Date:	
US Environmental Protection Agency		
Print Name	Date:	

#### 1.0 INTRODUCTION

The first tier of Pre-Record of Decision treatability testing is defined as Remedy Screening. The treatment objectives for this tier of testing are more qualitative than quantitative and the required quality assurance requirements reflect this less stringent approach. There is some flexibility for modification as the testing unfolds for reflective refinement in procedure or mixture to enhance understanding toward the overall objectives. The Quality Assurance Project Plan (QAPP) associated with this Work Plan has been prepared as a Category IV QAPP as designated in the "Guidance for Conducting Treatability Studies under CERCLA", EPA, October, 1992 and developed according to "Preparation Aids for the Development of Category IV Quality Assurance Project Plans", EPA, February, 1991.

#### 2.0 PROJECT DESCRIPTION

This project consists of Tier 1 testing to determine proof of principal (POP) for technologies recommended for treatment and prevention of acid mine drainage associated with a waste rock pile and pit lake for the Barite Hill Mine in McCormick County, South Carolina. A more detailed description for the project can be found in Section 1.0 of the Work Plan.

#### 3.0 QUALITY CONTROL ORGANIZATION (SOVEREIGN)

Table QA1 - Sovereign's Quality Control Project Team

Lee Josselyn, P.E.	Project Manager and Quality Control Manager
Jim Gusek, P.E.	Project Technical Lead – Senior Technical Reviewer
Paul Eger, P.E.	Field Quality Supervisor, Senior Technical Reviewer
Thomas Clark, Ph.D.	Laboratory Services Director – Lab QA/QC Manager
David Myers, P.E.	Project Engineer - Senior Technical Reviewer
Guadalupe Fattore	Project Engineer
Eric Wolaver	Project Engineer

#### 4.0 QUALITY ASSURANCE OBJECTIVES

The overall objectives of this project are to:

- Screen various source control technologies along with some metrics of their application characteristics that could be applied to the site to reduce the pyrite oxidation of the waste rock and contamination of the pit lake water.
- Screen several economical pit lake neutralization additives that may also provide some buffering capacity.
- Screen economical materials (local "clay" mixed with bentonite and a commercially available water block) for their potential as a flow barrier at the bottom of the pit.

These objectives are developed in detail in Section 3.0 of the Work Plan.

Test results to meet the Quality Assurance Objectives (QAO) have been classified as critical and noncritical. Critical results are those that are necessary to achieve project objectives. Noncritical results are those used for process control or general background readings. Table QA2 defines the results that will be obtained to assess the performance of the POP tests.

Table QA2 - Critical Nature of Test Results

OU1 Zone	Parameter	Method	Remediation Goal	Result
	pН	Hanna 9026 meter with combination electrode, Ag/AgCI	>5.5 for samples without alkalinity added >6 for samples with alkalinity added	Critical
	Metals	ICP-AES 6010C	~50% reduction	Critical
Waste Rock	Sulfate	EPA D516- 02/07- Turbidimetric	~50% reduction	Critical
Waste Rock	ORP	Hanna 9126 meter, Pt electrode with KCI electrolyte	Document the ORP at which the treatment is most effective	Noncritical
	Conductivity	SymPhony SP70 meter with epoxy probe	Document the Conductivity at which the treatment is most effective	Noncritical
	MPN	HACH, SRB- BART™	Document the MPN at which the treatment is most effective	Noncritical
Pit Lake	Coat and Tip Test (Failure ∠)	Procedure described in the Work Plan	Max. slope observed in the submerged pit floor (TBD)	Critical
Encapsulation	Falling Head Test	Method 9100	~K=1X10 <sup>-6</sup> cm/s	Critical
Pit Water	Alkaline Neutralization	Procedures described in	Best neutralization material will be evaluated based on: performance, quantity, availability, and cost	Critical
Treatment	Sulfate Reduction Neutralization	the Work Plan	Best carbon source will be evaluated based on: pH (5.5), lower ORP (<-100 mV), Fe <sup>+3</sup> (50% removal)	Critical

#### 5.0 APPROACH TO QA/QC

The purpose of QA/QC procedures is to produce data of known and expected quality by satisfying certain data quality indicators of precision, comparability, and completeness. This section serves to identify the QA/QC requirements for each testing group in the Work Plan. Below a summary of the activities in each testing group is provided along with its associated QA/QC requirements. Specifics on the group and individual tests, procedures, and analysis are provided in the Work Plan and SAP.

#### 5.1 Sample Characterization

**General Activity** - The initial sample characterization activity consists collecting some field data during the September sample collection site visit and sending initial samples out for processing (waste rock crushing) and base line analytical testing.

#### QA/QC Requirements

Review of field notes

- Review of chain of custody forms
- Review of stated procedures/methods to be used by the analytical laboratory
- Review of test results for precision, comparability, and completeness as well as documentation of analytical Lab QC procedures and methods used
- Surveillance of the lab practices may also be done (specifically for the rock crushing and blending phase)

#### 5.2 Waste Rock Testing

#### 5.2.1 Initial Qualitative Screening of Waste Rock

**General Activity** - Samples of the waste rock will be qualitatively (visually) tested when mixed with various/incremental doses of source control and neutralization compounds in a beaker. Testing parameters include color, odor, biofilm formation, observation of curds, solids, coatings, and noticeable changes and characteristics that might impact application techniques. Two different neutralization agents will also be mixed with the waste rock to determine how much agent is needed to create a pH of 7.0 and observe for noticeable changes and characteristics of the mix.

#### **QA/QC** Requirements

- A checklist/data sheet for testing will be developed and reviewed
- The laboratory technician will follow steps, enter data, log observations, take photos, etc. based on the checklist of criteria.
- Two laboratory technicians will be involved in the testing to confer observations and check notes for accurate description, time, and quantities that are used
- The data sheets and logs will be reviewed for comparability and completeness and agreement with test objectives

#### 5.2.2 Utilization Testing (USBM Modified Method)

**General Activity** – Potential source control amendments (with and without buffering) will be gravity flowed through a quantity of waste rock media to determine the amount of amendment "consumed" by the waste rock and make observations about the hydraulic characteristics.

#### QA/QC Requirements

- A checklist/data sheet for testing will be developed and reviewed
- The laboratory technician will follow steps, enter data, log observations, take photos, etc. based on the checklist of criteria.
- The data sheets and logs will be reviewed for comparability and completeness and agreement with test objectives

#### 5.2.3 Unsaturated Zone Amendment Screening

**General Activity** - Tests will be conducted in five-gallon buckets over numerous weeks, with various combinations of source control amendments and added alkalinity. The media will be rinsed with the rinsate collected and analyzed by parameters measurable with lab equipment and samples sent to an outside analytical laboratory.

#### **QA/QC** Requirements

- A checklist/data sheet for testing will be developed and reviewed
- The laboratory technician will follow steps, enter data, log observations, take photos, etc. based on the checklist of criteria.
- The data sheets and logs will be reviewed for comparability and completeness and agreement with test objectives
- · Review of chain of custody forms
- Review of stated procedures/methods to be used by the analytical laboratory

Review of test results for precision, comparability, and completeness as well as documentation of analytical Lab QC procedures and methods used

#### 5.2.4 Transition Zone Amendment Screening

**General Activity** - Tests will be conducted in five-gallon buckets over numerous weeks with various combinations of source control amendments and added alkalinity. The media will be submerged and rinsed with the rinsate and "surface" water collected analyzed by parameters measurable with lab equipment and samples sent to an outside analytical laboratory.

#### **QA/QC** Requirements

- A checklist/data sheet for testing will be developed and reviewed
- The laboratory technician will follow steps, enter data, log observations, take photos, etc. based on the checklist of criteria.
- The data sheets and logs will be reviewed for comparability and completeness and agreement with test objectives
- Review of stated procedures/methods to be used by the analytical laboratory
- · Review of chain of custody forms
- Review of test results for precision, comparability, and completeness as well as documentation of analytical Lab QC procedures and methods used

#### 5.2.5 Saturated Zone Amendment Screening

**General Activity** - Tests will be conducted in five-gallon buckets over numerous weeks with various neutralization and buffering agents with the media 100% saturated. Water samples will be collected and analyzed by parameters measurable with lab equipment and samples sent to an outside analytical laboratory.

#### **QA/QC** Requirements

- A checklist/data sheet for testing will be developed and reviewed
- The laboratory technician will follow steps, enter data, log observations, take photos, etc. based on the checklist of criteria.
- The data sheets and logs will be reviewed for comparability and completeness and agreement with test objectives
- Review of stated procedures/methods to be used by the analytical laboratory
- Review of chain of custody forms
- Review of test results for precision, comparability, and completeness as well as documentation of analytical Lab QC procedures and methods used

#### 5.3 Pit Lake Tests

#### 5.3.1 Pit Lake Floor Encapsulation

General Activity – Various quantities of clayey soil from the site, bentonite, and water (% moisture) will be mixed and tremied into water (5 gallon bucket or small fish tank) and the characteristics observed for spreading, settleability, coating, angle of repose, consistency, and failure slope. The same will be done for a commercially available water barrier product. The "best" performers will be tested for saturated conductivity in a falling head test.

#### **QA/QC** Requirements

- A checklist/data sheet for testing will be developed and reviewed
- The laboratory technician will follow steps, enter data, log observations, take photos, etc. based on the checklist of criteria.
- The data sheets and logs will be reviewed for comparability and completeness and agreement with test objectives

All calculations will be reviewed and approved.

#### 5.3.2 Pit Lake Neutralization

General Activity - Pit Lake Water will be titrated with several neutralizing (base) agents that have some buffering capacity. Several beakers will also be set up with Pit Lake Water and organic carbon sources (wood chips, limestone, manure, etc.) to evaluate the sulfate reduction potential to neutralize and buffer the water over numerous weeks.

#### **QA/QC** Requirements

- · A checklist/data sheet for testing will be developed and reviewed
- The laboratory technician will follow steps, enter data, log observations, take photos, etc. based on the checklist of criteria.
- The data sheets and logs will be reviewed for comparability and completeness and agreement with test objectives
- Calculations will be reviewed and approved.

#### 5.4 Data Quality Indicators

#### 5.4.1 Precision

Precision is the degree to which the analytical measurement is reproducible (i.e. that there is agreement between replicate measurements made under similar conditions for the same property). This is a measure of random error and can result from problems with sampling procedures, preservation, storage, shipment, preparation or analysis. Reproducibility among duplicate samples provides a determination of precision, which can be expressed as the relative percent difference in the amount of detected compounds between the original and duplicate samples. Relative percent difference (RPD) is quantified by the following equation:

RPD = 
$$\frac{(C1 - C2)}{(C1 + C2)/2}$$
 x100

where:

RPD = Relative percent difference

C1 = Larger of the two observed values

C2 = Smaller of the two observed values

Precision will be tracked by sending a duplicate samples to the analytical laboratory periodically. The duplicate will be a split of the sample collected at the same time and will not be conspicuously labeled as a duplicate.

#### 5.4.2 Comparability

Comparability is a qualitative term to describe the ability and appropriateness of taking two or more data sets to make collective conclusions.

The POP data will be compared to trends within the unit tests, against similar test units in the same family and to the control, and to the data obtained during the Initial Sample Characterization.

#### 5.4.3 Completeness

Completeness describes the percentage of valid data achieved versus what was planned by a measurement system. Most importantly, enough data should be generated to draw meaningful conclusions.

There are two components of data completeness: (1) the percentage of usable field samples taken of the samples planned; and (2) the valid (within QC objectives) data percentage of the total tests conducted.

For the purpose of these Tier one screening tests no specific % goal is provided. However as part of the data review the percentage of data completeness and usability will be evaluated and discussed with the project technical lead for acceptability in meeting test objectives.

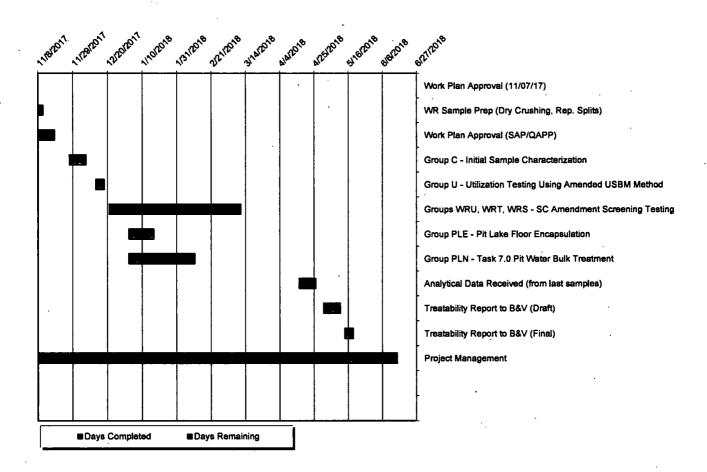
#### 6.0 QA FILES

Reviewer comments and initials will be maintained with the project files. Electronic project files will be maintained on a server with a data backup system. Original copies of logs, field notes, data sheets, laboratory results, chain of custody forms, etc. will be stored in a project file. A QA folder will be used for general QA files, lab procedures, surveillance documents, QA issues identification and resolution documents/log, and checklists showing that overall QA activities were done and the location of specific review and/or surveillance documents.

#### 7.0 REFERENCES

U.S. Environmental Protection Agency. Preparation Aids for the Development of Category IV Quality Assurance Project Plans. EPA/600/8-91/006 February 1991.

U.S. Environmental Protection Agency (Method 9100). Saturated Hydraulic Conductivity, Saturated Leachate Conductivity, and Intrinsic Permeability. Revision 0, September 1986.



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## SOVEREIGN CONSULTING INC.

## OPERATIONAL UNIT 1 (OU1) McCORMICK COUNTY, SOUTH CAROLINA

### **SAMPLING AND ANALYSIS PLAN (SAP)**

for

TIER 1 - REMEDY SCREENING
[PROOF OF PRINCIPAL (POP) TESTING]

DRAFT FOR REVIEW/APPROVAL

NOVEMBER 8TH, 2017

**SUBMITTED TO:** 

Black & Veatch 1120 Sanctuary Parkway, Suite 200 Alpharetta, GA 30009

**ATTACHMENT C** 

of the WORK PLAN

DV031.001

## **Table of Contents**

1.0	SITE BACKGROUND	. 1
2.0	SAMPLING OBJECTIVES	. 1
3.0	SAMPLING LOCATION, FREQUENCY AND ANALYSIS	. 1
3.1	Waste Rock Samples	. 1
4.0	SAMPLE DESIGNATION	. 2
4.1		
4.2	Resultant Material Sample Designation	. 2
5.0	SAMPLING EQUIPMENT AND PROCEDURES	. 2
6.0	SAMPLE HANDLING	. 2

#### **ATTACHMENTS**

ALIA	CHIVII	ENIS
SAP1	C1	Initial Sample Characterization - Check List
	C2	Initial Qualitative (Beaker) Screening - Data Sheet
	C4	Source Control Treatment Testing - Lab Analysis
	C5	Source Control Treatment Testing - Bench Parameters
	C6	Pit Lake Encapsulation - Coating Evaluation - Data Sheet
	C7	Pit Lake Encapsulation - Falling Head Test - Data Sheet
	C8	Pit Water Treatment - Titration - Data Sheet
	C9	Pit Water Treatment - Sulfate Reduction - Bench Data Sheet
	C10	Pit Water Treatment - Sulfate Reduction - Analytical Data Sheet
SAP2	Modifi	ed USBM Procedure

#### 1.0 SITE BACKGROUND

The Site background is included in Section 1.1 of the Work Plan.

#### 2.0 SAMPLING OBJECTIVES

"The purpose of the SAP is to ensure that samples obtained for characterization and testing are representative and that the quality of the analytical data generated is known. The SAP addresses field sampling, waste characterization, and sampling and analysis of the treated wastes and residuals from the testing apparatus or treatment unit." (Section 3.6, Guidance for Conducting Treatability Studies under CERCLA. USEPA, 1992). Because this is tier one testing (screening process) the required results are more qualitative (pass/fail) than quantitative. Thus this Sampling and Analysis Plan is related to characterizing the materials collected for application of the Proof of Principal (POP) testing including describing the testing/methods and data management in enough detail to demonstrate satisfaction of the test objectives. Overall to support decisions related to remedy selection and further testing. Note that this level of testing requires some latitude for adjustment (changes, additions, or omissions) as developments are made that may impact the objectives of the test.

Overall and specific objectives for each testing phase (group) are provided in the Work Plan, Section 3.0.

#### 3.0 SAMPLING LOCATION, FREQUENCY AND ANALYSIS

There are eight groups of tests in the work plan including the initial material characterization, five progressive test groups for waste rock/ARD amendment evaluations, and two groups for pit lake testing for neutralization and floor encapsulation agents. Test results for each group will contain some combination of observations, "local" measurement (using hand held probes and HACH type tests), and outside laboratory analytical work. Individual tests in each group are identified in the Work Plan Tier 1 Matrix along with details about each test (type, constituents, amounts, application, objectives). Tables (data sheets) set up for the SAP are an extension of the matrix that identify specific data that needs to be acquired and samples to be taken for each test unit. The SAP data sheets are to be used for data entry, data management, and for check lists for completeness (sample location/source, frequency, and analysis). Data sheet for each test group are identified in Table SAP1 below. Data sheets and check off lists are provided in Attachment SAP1 Data Sheet Tables and SAP2 USBM modified Procedure

#### 3.1 Waste Rock Samples

Table SAP1 - Data Sheets for Each Test Group

	Test Group	Test No.	Attachment SAP1 Data Sheet Table
Field Samples (WR & Pit)	Initial Sample Characterization	C (1-4)	C1
	Initial Qualitative (Visual) Beaker Screening	IQ (1-5)	C2
Marks Book	Utilization Testing (modified USBM method)	U (1-6)	Attachment SAP3
Waste Rock	Unsaturated Zone	WRU (1-7)	C4 & C5
	Transition Zone	WRT (1-5)	C4 & C5
	Saturated Zone	WRS (1-3)	C4 & C5

		2		
	Floor Encapsulation		PLE (1-11)	C6 & C7
Pit	Floor Encapsulation	PLE (12 -17)	C0 & C7	
rii.	Water Bulk Treatment		PLN (1-4)	C8, C9, &
			PLN (5 -26)	C10

#### 4.0 SAMPLE DESIGNATION

Samples will be collected from the raw/feed material (influent or initial condition of the water and soils to be used in the test) and from the effluent/resultant materials (produced from the testing application). Section 4.1 and 4.2 describe the sample designation for the feed and resultant material.

#### 4.1 Feed Material Sample Designation

The raw and processed materials used as the base testing materials (prior to testing application) will be analyzed once in the initial characterization phase and as solutions/surrogates are made up for application in specific tests. These tests are indicated as "influent" samples on the data sheets and are given a sample number unique to the sample unit or group (described below). The analysis for the feed material will be reused for each test unit unless there is good reason to believe the characteristic of the material has changed.

#### 4.2 Resultant Material Sample Designation

The treated, effluent, or manipulated materials from each test will be considered the "resultant" materials of the testing. Individual samples of the resultant materials will be identified first by the test group, then by the specific test number being conducted, and the date. The test group and specific test numbers are provided in the SAP Data Sheets and in the Work Plan Matrix

For example sample designation: WRU1-103017

WRU = Test Group = Waste Rock, Unsaturated Zone

1 = 1st specific test unit (identified in the matrix)

103017 = Sample collection date of October, 30th, 2017

Samples that will be collected weekly will have the same group, same test number but a different date. If multiple samples are taken on the test unit the same day then the sample numbers will have a, b, c, d, etc. added after the date to reference back to the data sheet and chain of custody form.

#### 5.0 SAMPLING EQUIPMENT AND PROCEDURES

Details on specific equipment and procedures are provided in the work plan, work plan matrix, and SAP data sheets.

#### 6.0 SAMPLE HANDLING

Chain-of-Custody forms identifying all the sample designations (numbers), containers, analysis to be done, and other field data required by the laboratory will be completed and accompany each sample container to the off-site laboratories. Samples will be prepared/packaged as per the receiving laboratories requirements. Water samples will be filtered, preserved (if required), and sealed in the lab sample bottles. Soil samples will be sealed in double Ziploc type bags.

The majority of the water and solid samples will be analyzed/characterized by Solfatara. Metal analysis by Inductively Couple Plasma Atomic Emission Spectrometry (ICP-AES) will be conducted by Colorado School of Mines (CSM) Chemical Laboratory. Solid samples will be sent to an off-site laboratory for Atterberg Limits and ABA testing.

## **Initial Sample Characterization - Check List**

Table C1

					Field	Data							Lab (	hara	cteriz	atior	1			
		Sample	Plant Type	Vegetative Density	Photos	Depth	Visual Charac.	Material Size	Specific Gravity	Atterberg Limits	Sieve Analysis	Field Capacity	Res. Acidity	ABA	dol	Hd	ORP	Sulfate	Specific Conductivity	Alkalinity
Test	Ð	Cover Plants	Х	X	Х															ш
C2	1 👼	Borrow Soil			X	X	Х	Х	Х	×	X	×								ш
C3	] ທ	Waste Rock			Х	Х	Х	X	Х		Х	X	X	X	X	X				
C4	1	Pit Lake			Х	Х	Х								Х	Х	X	X	Х	Х
C5	C5 February Page 1	Well BH26				Х									X	Х	Х	X	Х	Х
C6	] =	Well BH28				X									×	Х	X	X	X	Х

#### Initial Qualitative (Beaker) Screening - Data Sheet Table C2 Sampler(s) Initials: Finish Time: Observe the characteristics and extent of change of the subject material mixed with incremental amounts of amendment. Evaluate the **Objective** approximate dose that may be used for further testing (pH of 7.0 for caustics). Look for favorable or adverse effects that may guide or limit application techniques and effectiveness of the amendment. [circle the work plan test # that applies] (alternative?) **Test ID** Test# IQ1 IQ2 IQ3 IQ4 IQ5 Subject Material Waste Rock Waste Rock Waste Rock Waste Rock Waste Rock Amendment: Whey Limestone SLS Bkg Soda **Initial Condition** photo? item conc. volume moisture description/notes mass Subject Material Waste Rock Amendment Descriptions: changes/characteristics (color/odor/consistancy)(presence of biofilm/curds/coating/precipitate/separation/etc.)(faverable/adverse properties?) amount of amendment added 1st Increment how added time: \_\_\_\_\_ 2nd increment photo? amount of amendment added how added time: 3rd Increment amount of amendment added how added time: \_ pH: 4th Increment amount of amendment added how added pH: time:

[Only one test ID per sheet. Use additional sheets as as necessary for more increments/notes]

QC intls/data
---------------

Source Control Tr	Ta	Table C4			
Sampler(s) Name(s): Sampler(s) Initials:		Sampling Start Time: Sampling Finish Time:	<u>·</u>	Date:	<u> </u>
Objective To demonstrate a	id generation suppression in waste rock treated with source control amendments				

				Lab Analysis				ICF	-AES Resu	ılts			
	Test	Treatment	Frequency	ICP-AES'	Al	As	Cd	Cu	Fe	Mn	Ni	Pb	Zn
		Waste Rock -	Unsaturated Z	one					mg/L				
	WRU1	POP Control		4					I				
	WRU2	POP-SLS w/Alk		4					L				
	WRU3	POP-Milk		42									
	WRU4	POP-Milk w/Aik		4									
	WRU5	POP-Whey w/Alk	Monthly	4									
ø	WRU6	POP-SLS w/Alk		4									
Reactors	WKU6	then Milk w/Alk				<u> </u>			l	L			
		POP-SLS w/Alk		4									
_	WRU7	then Whey		4			1						
Effluent from		Waste Rock	- Transition Zo	ne		•							-
ב	WRT1	POP Control		4			I						
6	WRT2	POP-SLS w/Alk		42					l			l	
₽	WRT3	POP-Milk	Monthly	4									
Ш	WRT4	POP-Milk w/Alk		4									
	WRT5	Pop-Whey		4									
		Waste Rock	- Saturated Zo	ne									
j	WR\$1	POP Control		4									
}	WRS2	POP-Alk & Milk	Monthly	4									
	WRS3	POP-Alk & Whey		42									
		Total No. of San	nples	63									

Note: (1) ICP Metals: Al,As,Cd,Cu,Fe,Mn,Ni,Pb,Zn

(2 )Duplicates will be sent to CSM for QA/QC

Source Control Treatment Testing - Bench Parameters	Table C5				
Sampler(s) Name(s): Sampler(s) Initiats:	Sampling Start Time: Sampling Finish Time:	Date:			
Objective To demonstrate acid generation suppression in waste cock and oil lake water treated with so	surce control amendments				

.

_			Frequency	No. of		Fie	ld Paran	neters		Observations			
[	Test	Treatment	Frequency	Samples	рН	Conductivity		Sulfate	MPN	Color/Odor	Algae Growth/Precip. Crust	Photos	
						Waste Roc	k - Unsai	urated Zone		•			
	WRU1	POP Control		16									
	WRU2	POP-SLS w/Alk		16									
	WRU3	PÖP-Milk	Weekty (16 weeks)	16									
	WRU4	POP-Milk w/Alk		16						l			
	WRU5	POP-Whey w/Alk		16									
គ្គ	WRU6	POP-SLS w/Alk then Milk w/Alk		16									
Reactor	WRU7	POP-SLS w/Alk		16				-		1			
E		then Whey		<u> </u>	<u> </u>	14/4- 5-		-101 7		L			
Effluent from					,	Waste Ko	CK - I Fan	sition Zone					
Ħ	WRT1	POP Control	Weekly (16 weeks)	16									
<b>3</b>	WRT2	POP-SLS w/Alk		16						ļ		ļ	
馬!	WRT3	POP-Milk		16									
- 1	WRT4	POP-Milk w/Alk		16									
ļ	WRT6	Pop-Whey		16	<u> </u>							<u> </u>	
١,	Waste Rock - Saturated Zone												
	WRS1	POP Control	Weekly	16	↓								
- 1	WRS2	POP-Alk & Milk	(16 weeks)	16									
-	WRS3	POP-Alk & Whey		16								L	
L		Total No. of Sam	pres	240	240	240	240	240	240	J			
											•		
												•	
											•		
												•	
						•							

Pit Lake Encapsulation - Coating Evaluation - Data Sheet Table C6					6									
Sampler(s) Name(s):			Sampling Start Time:					Date:				_		
Sampler(s)	Initiats:					Sampling i	Inish Time:						-	
Objectiv	Objective To conduct trial and error tests to identify a soll, bentonite, and water content "grout" that would distribute on					ntent "grout" that would distribute on	pit lake fi	oor to see	lit, and n	ot slump too	much on ste	oper submerged slopes, if present.		
					Barrier	Material (	Quantity						Tipped T	est
Test	Material Tested	Soil (g)	Water (ml)	Bentonite (9)	Date	Time (Start)	Time (End)	Observations	Max Cling 4	Repose2	Failure 2	Date	Dur. of Test (hr)	· Observations
PLE1	Borrow Soil													
PLE2	Borrow Soll												Ì	
PLE3	Borrow Soll												1	
PLE4	Borrow Soll												ŀ	
PLE5	Borrow Soil												Ì	
PLE6	Borrow Soil												24	
PLE7	Borrow Soil												1	
PLE8	Borrow Soil	1											1	
PLE9	Borrow Soil	1											1	
PLE10	Borrow Soil	1											1	
PLE11	Aquablok	1											1.	
Duplica		-												
	Material	I			Barrier	Material (	Quantity						Tipped T	est
Test	Tested	Soll	Water	Bent.		Time	Time				_		Dur. of	
	162160	(g)	(ml)	(g)	Date	(Start)	(End)	Observations	F∠	R∠	C∠	Date	Test (hr)	Observations
PLE12	Borrow Soil												24	
Combi	nation											•		
	Material	I			Barrier	Material (	Quantity						Tipped T	est
Test	Material Tested	Soil (g)	Water (ml)	Bent. (g)	Date	Time (Start)	Time (End)	Observations	F∠	R∠	C∠	Date	Dur. of Test (hr)	Observations
PLE13	Borrow Soil												24	

Pit La	ike Encapsu	lation - Fallin	g Head Test	- Data Sheet		Table C7		•		
Sampler(s)			- -	Sampling Start Time: Sampling Finish Time:	· · · ·					
Objectiv	/8	To conduct trial and error tests to identify a soil, bentonite, and water content "grout" that would distribute on pit lake floor to seal it, and not stump too much on steeper submerged stopes, if present.								
•				Falling Head 1	ests (Method 9100)		-	<del></del>		
Test	Material Tested	L	a	Α	t	h <sub>o</sub>	h <sub>1</sub>	Observations		
Borrow Soil										
PLE12	Borrow Soil									
PLE13	Borrow Soil									
PLE14	Borrow Soil									
PLE15	Borrow Soil									
PLE16	Borrow Soil									
PLE17	Aquablok	<u> </u>				<u> </u>				
Where:	test will be conducted									
	L = The length of									
		tional area of the st								
		ctional area of the s	pecimen, L*							
	t = Elapsed time f	rom ho to h <sub>1</sub> , T								
	ho = Initial Height	of water								

 $h_1$  = Final height of water Hydraulic Conductivity

K = <u>2.3 a</u>L

At

Log<sub>10</sub> ho

## Pit Water Treatment - Titration - Data Sheet

Sampler(s) Name(s):

**Table C8** 

Sampler(s) Initials:		_						
·								
Objective								
		•						
	[Circle the chemical that applies]							
Test ID	PLN1	PLN2	PLN3	PLN4				
Subject Material	Upper Pit Lake Water	Upper Pit Lake Water	Upper Pit Lake Water	Upper Pit Lake Water				
Chemical	Caustic Soda	Lime	Quicklime & Fine Limestone	Limestone				

Sampling Start Time:

	Titration								
Added (mg)	Cumulative (mg)	pH							
	l	I .							

Note: The titration test will be conducted once for each chemical

FILT	rater ireau	nent - Sun	ate Reduction	ite Reduction - Bench Data Sheet					
Sampler(s Sampler(s	i) Name(s): i) Initials:		_ Sarr	npling Start Time: pling Finish Time:		Date:		· ·	
Objecti	ve	To determine desi	gn parameters for future	passive treatme	nt testing.			•	
		Monitoring		Carbon	FI	eld Parame	eters		
Test	Material Tested	Frequency	Carbon Source	Required (mg)	рН	ORP	Sulfate	Observations	
PLN5		T							
PLN6						Ļ	<b></b>		
PLN7							<del> </del>		
PLN8			<b>—</b>			<del> </del>	<b>_</b>		
PLN9 PLN10						<u> </u>			
PLN11						<u> </u>	<del>                                     </del>		
PLN12		ļ				<u> </u>	<del>                                     </del>		
PLN13		İ				<u> </u>			
PLN14		ļ							
PLN15	Upper Pit	Weekly							
PLN16		(8 Weeks)							
PLN17							<u> </u>		
PLN18		1				Ļ			
PLN19		1					ļ		
PLN20		1				ļ			
PLN21		l	-				<b></b>		
PLN22 PLN23					-	<del>                                     </del>	<del>                                     </del>	-	
PLN24			<del>                                     </del>			<del>                                     </del>			
PLN25					<del>                                     </del>	<del>                                     </del>	1	<del>                                     </del>	
NEO	1	1			L	L		<u></u>	

Total No. of Samples

### Pit Water Treatment - Sulfate Reduction - Analytical Data Sheet

Table C10

Sampler(s) Name(s): Sampler(s) Initials:	Sampling Start Time: Date: Sampling Finish Time:
Objective	To determine design parameters for future passive treatment testing.

			Observations								
Test	Material Tested	Al	As	Cd	Cu	Fe	Mn	Ni	Pb	Zn	
PLN5				<del> </del>					 		
PLN6				.†	<u> </u>						
PLN7											
PLN8											
PLN9											
PLN10				<u> </u>	Ļ	<u>.</u>					
PLN11			_	1					ļ		
PLN12				ļ					ļ		
PLN13				<del> </del>					<del></del>		
PLN14 PLN15	Linnas Dit			+	ļ						
PLN15	Upper Pit Layer Water	_		<del>-{</del>					<u> </u>		
PLN17	Layer Water										
PLN18				<del>                                     </del>	<u> </u>						-
PLN19				† — · · · · ·							
PLN20				Î							
PLN21											
PLN22											
PLN23											
PLN24				ļ							
PLN25											
PLN26					<u> </u>						

Notes:

<sup>(1)</sup> ICP (Al,As,Cd,Cu,Fe,Mn,Ni,Pb,Zn) testing will be conducted only once at the end of the testing (week 8)

## SCREENING PROCEDURE FOR THE CONSUMPTION OF BACTERICIDES AND DAIRY AMENDMENTS

Portions Excerpted from Kleinmann and Erickson, USBM RI 8847, 1983. (MODIFIED)

#### **Bactericide (SLS) Consumption Screening Procedure**

<u>Background</u> - If, based on general visual and analytical considerations, the site or material appears appropriate for the surfactant treatment (i.e., un-oxidized pyrite is present), the obvious question is how much surfactant is required. The concentrations needed to kill *Acidothiobacillus ferrooxidans* in small volumes of pyritic mine waste are quite low (25 to 40 mg/L). Applying surfactant at these concentrations would only be partially effective at an actual mine site because it would fail to reach most of the oxidizing pyrite at the strength required. This is partially due to the degradation of the surfactant by the residual acidity in the waste, probable adsorption to mineral surfaces and other factors. Consequently, it is necessary to determine the "degradation" or sorptive capacity (consumption) of the material being treated and to compensate for those factors with either extra surfactant or an additive that can improve the effective distribution (such as pre-wetting or a buffering reagent to neutralize the acidity).

<u>USBM Method</u> - A fairly simple laboratory procedure that was developed to provide an estimate of adsorption potential for overburden and mine waste for surfactant applications. A representative sample of the material to be treated is placed in a large, tared Buchner funnel, tamped to a uniform depth of 2 in, and weighed. A surfactant, such as SLS, is applied evenly over the material at approximate loads of 60 mg SLS/kg of mine waste. Typical SLS concentrations range from 300 to 30,000 mg/L and are selected on the basis of the anticipated infiltration rate and the field capacity volume of the sample mass.

The primary goal of this screening test is to determine SLS consumption by adding a precise amount of SLS to a sample of mine waste and then rinsing the sample until all available or unused SLS is recovered. The amount of initial SLS added (in mg) will be a function of the dry sample mass, its field capacity, and the desired initial unit SLS content (baseline is 60 mg SLS/kg waste). In the USMB method the rinsed SLS concentration was determined for every 200ml collected. For the modified test used for this project a Baseline Solution Application Worksheet (in Excel) has been developed for this purpose that can quickly be used to approximation of concentration of remaining SLS by setting up comparison "standards" for volume and decay time of foam produced and conductivity.

<u>Modified Method</u> - If the dry sample receives the field capacity volume that is infused with SLS, it is safe to assume that all the initial SLS it receives is retained within the sample until the first rinsing volume is delivered. Note: the rinsing volume should be equal to or greater than the field capacity volume for the sample mass.

After the surfactant application, the top section of a second funnel or a perforated plate is fitted onto the packed funnel to allow even distribution of a distilled water rinse.

The rinse solution is continuously applied through plastic tubing from a distilled water reservoir. The height of the reservoir is adjusted to balance influent and effluent flow rates; in relatively impermeable samples, inundation is unavoidable and effluent flow rates can be as slow as 100 mL/h. Rinse effluent is collected in 100-mL fractions until no sudsing is observed on shaking.

<u>SLS Concentration Standard Development</u> - For rapid Tier 1 testing of relative SLS consumption, eight standard solutions of SLS will be prepared:

100 mL of each SLS standard solution will be subjected to rapid and vigorous mixing in a high speed magnetic stirrer or similar device to generate foam as a means of objectively and consistently assessing sudsing. The stirring time and stirring speed will be held constant for this standardization step. Foam volumes from each standard solution will be measured to the nearest 25 mL in a graduated container and Table 1 below will be generated. As the SLS concentration falls, the time required for the foam to decay to the original 100 mL of standard solution will decrease as well. This foam decay time data will also be observed and recorded to the nearest minute. From experience, the variations in foam decay time will be more indicative of SLS concentration than foam volume.

Conductivity of the SLS solutions will also be recorded to evaluate its ability to be used as a standard for SLS concentration.

Table A – SLS Concentration Standards Development

SLS Concentration	Foam Volume (mL)	Time for 100 mL liquid to reform (min.)	Specific Conductance (µS)
2%			-
1%			
0.5%			
0.1%			
0.05%			
0.025%			
0.01%			

Other standard solutions may be prepared as needed.

#### Field Capacity Test

The field capacity needs to be calculated to estimate the volume of SLS solution that will be applied to saturate the waste rock initially. Overshooting the field capacity by 10-20% may be necessary to ensure good uniform saturation of the solution in the 2 inches of media but overshooting significantly will reduce the accuracy of the mass balance calculation for amount "consumed"

1)	Place about 1,200 grams of waste rock into a microwavable container - Weigh g
2)	Dry in oven or microwave (no more than 5 min intervals on 20% power) Weigh g
	Repeat drying step until weight is constant – record g (dry mass)
3)	Calculate water loss [(2) - (4)] = g
4)	Calculate moisture content (water loss/dry solids) [(3) / (2a)] =%
5)	Weigh empty Buchner funnel and qualitative filter paper g Mark 2" line above filter level.
6)	Place/tamp 2" of dry sample in Buchner funnel on top of filter paper (position the funnel in a 1,000 mL beaker so it is stable
7)	Weigh filter + solids = g
8)	Add 500 mL of distilled water, cover filter to minimize evaporation and wait about 2 hours for the water to bleed through
9)	Measure vol. or mass of water bled-through + any water remaining on top (decant off) mL or g; measure pH and conductivity
10	) Weigh funnel + solids + retained waterg

		water retained [(1		g = ml; compare to	o calculated
water retained	n007] t	ıl – (9)] =	ml.		
12) Calculate ma	ess of dry solids [(7	7) - (5)] =	.g		
13) Calculate fiel	d capacity (mass	water / mass dry s	soil) [(11) / (12)] =	%	
			, . , , , , , , , , , , , , , , , , , ,		
Relative Hydraulic Co					
The relative hydraulic	_		-		
this testing and evalu					
Buchner filters the so	olid sample size m	ust be reduced to	leave space for se	olution on top of t	he solids.
· · · · · · · · · · · · · · · · · · ·	-	Iter surface in the	•	•	
2) Place/tamp 50	00 g of dry solids i	in a Buchner funne	el on top of a qual	itative filter paper	About 2 –
2.5 cm) and n	neasure depth of t	the solids m	ım		
3) Measure 100	ml of the test solu	ution and quickly p	our into Buchner	funnel. Note/mar	k solution
level from lip	of funnelmm	, and start time			
4) Note when so					
		y slow note the so		every 15 minutes	until aone
•	15 min m	•	idilon lovo, y		u 900
	30 min mi				
	45 minm				
5) Calculate sat	urated nydraulic d	conductivity (K)	mm/min then	/ 60,000 =	_ m/s
Results Table – Rela	tive Hydraulic Cor	nductivity			
Solution	Distilled Water	SLS solution	Milk Soln	Whey Soln.	]
% soln	0.0%				]
Hyd Cond. (m/s)					]

#### **SLS Consumption Tests**

Four tests are planned to evaluate the SLS degradation or sorptive capacity of the waste rock using SLS alone and SLS with three different concentrations of NaHCO<sub>3</sub>. Concentrations to be determined from the beaker tests.

#### For each test:

- A representative sample of (dry) waste rock is placed in a large Buchner funnel and tamped uniformly to a depth of 2 in
- Mass of the funnel and funnel + solids is recorded
- A solution of SLS is prepared and applied evenly over the material at a volume approximated by the field capacity. (The SLS concentration is initially established using 60 mg SLS/kg waste as the goal and then adjusted if needed during the test.)
- Mass of the funnel + solids + retained solution is recorded along with the volume of solution applied and recovered.
- 100 ml of rinse water is then applied over the material and recovered. The resulting solution is then
  - o Measured for volume, pH, and conductivity
  - Run through a foaming test for estimation of remaining SLS concentration (using the same methods that were used for establishing the standards)
  - o Retained for combining with all recovered water samples

#### **ATTACHMENT SAP2**

#### Foaming Test Evaluation Process

If the no sudsing is observed after a single rinse, the initial SLS mass was considered fully consumed and the test needs to be repeated (using a fresh, dry sample) with a higher initial SLS content.

In contrast, if a full liter of rinsing solution produces sudsing, one can infer that the initial SLS content was too high and the test needs to be repeated (using a fresh dry sample) with a significantly lower initial SLS content.

In this way, the initial "target" SLS content which still yields sudsing can be bracketed. It is assumed that a solution that sudses will still contain enough SLS to provide bactericide characteristics. This will be evaluated during the initial SLS standard development step.

Once a successful SLS concentration has been bracketed (sudsing is observed after the first rinse but not after the 10<sup>th</sup> rinse) then combined volume of recovered solution/rinsate will be mixed and a sample submitted for anionic surfactants analysis (concentration) by the methylene blue technique.

#### **Pre-Wetting Test**

To assess the effects of initial moisture content, the test will repeated with the solids sample pre-wetted to its field capacity prior to delivering the SLS solution. The subsequent recovered solution will also be composited and submitted for analysis by the methylene blue technique.

#### **Data Tables**

Table 1A – SLS Consumption Test Set-up and SLS Application Data

Parameter	Initial Test	Re-adjusted SLS conc. (if needed)	Re-adjusted SLS conc (if needed)	Pre-Wetting Test
Test Unit Number	-			
SLS % solution				
Test Date				
Test Start Time	·			
Test Person (initials)				
Mass Funnel (F)				
Mass F+Solids (S)			·	
Vol pre-wet water applied				
Vol SLS soln applied				
Vol SLS soin recovered				
Mass F+S+retained SLS soln				
Selected for methylene blue				

Tables 1B - Sample Rinsing Data Sheets - SLS only

Test Uni	t Number:	SL	S Conc:	T	est Date:	St	Start Time:		
Rinse #	Rinse volume (ml)	Recovered volume (ml)	рН	Cond. (m\$/cm)	Foam Volume (mL)	Decay time (min.)	Est. SLS Conc (mg/L)	Est. SLS mass (mg)	
1	100 mL								
2	100 mL			•		-			
3							I		
4									
5									
6									
7					· .			•	
8							•		
9									
10									
TOTAL									

For subsequent SLS concentration adjustment tests and/or Pre-wetting test

Test Uni	t Number:	SL	S Conc:	T	est Date:	St	Start Time:	
Rinse #	Rinse volume (ml)	Recovered volume (ml)	рН	Cond. (mS/cm)	Foam Volume (mL)	Decay time (min.)	Est. SLS Conc (mg/L)	Est. SLS mass (mg)
1	100 mL		-					
2	100 mL							
. 3								
. 4								
5								
6					•		Ì	
7	_							
8								
9								
10								
TOTAL								

For subsequent SLS concentration adjustment tests and/or Pre-wetting test - use add'l pages as needed

Test Uni	t Number:	SL	S Conc:	T	est Date:	St	Start Time:	
Rinse #	Rinse volume (ml)	Recovered volume (ml)	рН	Cond. (mS/cm)	Foam Volume (mL)	Decay time (min.)	Est. SLS Conc (mg/L)	Est. SLS mass (mg)
1	100 mL			· <del>-</del>				
2	100 mL							
3					•			
4								
5			_					
6								
7								
8								
9								
10								
TOTAL								

#### Set Up for SLS + Baking Soda (NaHCO<sub>3</sub>) tests

SLS degradation is calculated as the difference between the mass of SLS applied to the sample and that eluted and it will be recorded as milligrams of SLS consumed per kilogram of sample. Alternatively, degradation can be reported as pounds SLS per acre inch, calculated from the known funnel area and sample thickness. The laboratory test provides only a relative measure of degradation capacity since field conditions are not duplicated.

Once the degradation mass is estimated, a second round of baking soda (NaHCO<sub>3</sub>) buffered SLS solution applications to three duplicate samples of mine waste will be conducted.

In this testing step, a series of buffered SLS solutions will be prepared in accordance with the findings of previously-conducted beaker tests from which the concentration of baking soda required to neutralize the stored acidity in the mine waste to pH 7 will have been determined.

Foaming/sudsing tests will be conducted on rinsates from three identical dry waste rock samples exposed to three concentrations of baking soda with SLS concentration held constant (at say 2X the "degradation loss concentration" as determined above). The data in Tables 3 to 5 will be entered and the final rinsate samples will be submitted for methylene blue testing for confirmation.

The pre-wetting test will not be conducted for these three test units

#### **Data Tables**

Table 2A, 3A, 4A – SLS Consumption Test Set-up and SLS + NaHCO₃ Application Data

Parameter	Initial Test	Re-adjusted SLS conc. (if needed)	Re-adjusted SLS conc (if needed)
Test Unit Number			_
SLS % solution			
NAHCO <sub>3</sub> (mg/l)			
Test Person (initials)			
Test Date			
Test Start Time			
Mass Funnel (F)			
Mass F+Solids (S)			
Vol pre-wet water applied			
Vol SLS soln applied	* - *		
Vol SLS soln recovered			
Mass F+S+retained SLS soln			
Selected for methylene blue			

Table 2B, 3B, 4B - Sample Rinsing Data Sheets - SLS + NaHCO<sub>3</sub>

Unit Number:		SLS %:	NaHCO	3 (mg/l)	Test Date:		Start Time:	
Rinse #	Rinse volume (ml)	Recovered volume (ml)	рН	Cond. (mS/cm)	Foam Volume (mL)	Decay time (min.)	Est. SLS Conc (mg/L)	Est. SLS mass (mg)
1	100 mL							
2	100 mL							
3								
4								
5							-	
6								
7								
8								
. 9								
10					-			
TOTAL								

For subsequent SLS concentration adjustment tests and/or Pre-wetting test

Unit Number:		SLS %:	NaHCO	3 (mg/l)	Test D	Test Date:		me:
Rinse #	Rinse volume (ml)	Recovered volume (ml)	рH	Cond. (mS/cm)	Foam Volume (mL)	Decay time (min.)	Est. SLS Conc (mg/L)	Est. SLS mass (mg)
1	100 mL							
2	100 mL					_		
3								
4					•			
5								
· 6								
7								
8								
9				_			•	
10								
TOTAL								

For subsequent SLS concentration adjustment tests and/or Pre-wetting test – use add'l pages as needed

Unit Number:		SLS %:	NaHCC	)3 (mg/i)	Test D	Test Date:		me:
Rinse #	Rinse volume (ml)	Recovered volume (ml)	рН	Cond. (mS/cm)	Foam Volume (mL)	Decay time (min.)	Est. SLS Conc (mg/L)	Est. SLS mass (mg)
1	100 mL							
2	100 mL							
3				Ī				
4								
5								
6								
7								
8								
9			•					
10	•							
TOTAL								

The data from this phase of the testing will be used to determine the reagent concentrations that will be used in the subsequent tests.

#### Equipment & Materials

- 15 kg of mine waste
- SLS powder (200 grams or as needed)
- Baking soda (200 grams or as needed)
- Magnetic stirrer
- 500 mL graduated cylinder (foam volumes)
- Balance (1kg cap.)
- pH & conductivity probes
- 4 each large Buchner funnels

- 8 each 1 liter bottles (for std SLS solutions)
- 10 each 250 mL beakers (for SLS make-up, rinsate collection)
- 1 each bucket for combining all recovered (pass through) solutions
- 5 each sample bottles for methylene blue testing
- DI water (4 liter reservoir)
- •

#### **Milk Consumption Protocol**

<u>Background</u> - Exposing milk (a colloidal suspension of casein, whey proteins, and lactose) to acidic, pyrite-bearing mine waste should result in the separation of curds and whey. The "consumption" of curds is expected to be proportional to the micro-encapsulation of exposed pyrite grain surfaces in the unsaturated mine waste, creating an anaerobic microenvironment for desired microbial activities. This encapsulating layer has been proven to provide a substrate for heterotrophic microbes to out-compete *Acidothiobacillus ferrooxidans* under saturated conditions. It may also work in unsaturated conditions.

#### Milk Relative Hydraulic Conductivity and "Consumption" Tests

The following protocol can be used or modified as needed to quantitatively estimate how milk behaves when exposed to acidic mine waste.

1)	Prepare 200 mL of non-fat milk using powdered milk: 190 mL tap water + 19.3 grams
	powdered milk – this can also be diluted to a lesser strength%
	mL tap water + grams powdered milk
2)	Run the Relative Hydraulic Conductivity test with the milk solution. It will be very
	important to record the solution level over time with milk because it is expected to cause
	plugging (curds). The change in Hyd. Cond. over time will be calculated to evaluate
	results
	a. Wait until all the milk solution bleeds through sample into beaker; if it doesn't, (in
	2 hrs) stop the test – sample is plugged with curds. Note time hrs
	levelmm
3)	Collect bleed through solution, measure volume (A) cm and pH Retain
	separately.
4)	If all the milk has bled through add another 100ml of milk solution and record level over
	time again
	a. 15:00cm
	b. 30:00cm
	c. 45:00cm
	d. 60:00cm
	e. Wait until all the milk solution bleeds through sample into beaker; if it doesn't, (in
	2 hrs) stop the test – sample is plugged with curds. Note time hrs
	levelmm
5)	Collect bleed through solution, measure volume (B) cm and pH
6)	If the pH of the bleed through is <4.5 (curds should still form)
	a. Repeat with another 100ml of milk solution. Tests C and D (then stop)
7)	Calculate relative hydraulic conductivity (K) for each 100 ml test
	a m/s
	b m/s
	c m/s
	d m/s
8)	Place 20ml of bleed through solution form each test in to separate 25 mL graduated
	cylinders. Add 20 drops of hydrochloric acid to each; wait four hours and measure
	amount of curds (solid white) and whey (cloudy solution)
	a mL curds mL whey
	b mL curds mL whey

	c mL curds mL whey
	d mL curds mL whey
9)	Compare values with "baseline" non-fat milk 11 mL curds; 12 ml whey.
10)	Proportionally estimate remaining "unreacted" milk from the original milk volume
	ag
11)	Optional - rinse the sample with deionized water to determine if milk proteins (which are

#### **Equipment & Materials**

- 0.5 kg mine waste
- Microwave oven
- 2 each 250 mL glass beakers

soluble at neutral pH) can be mobilized.

- pH meter
- Buchner funnel & paper filter
- Balance (1kg cap.)

- Ruler (cm)
- Clock
- Muriatic acid (31% HCl)
- Powdered milk (100 grams)
- Tap water (500 mL or as needed)

#### **Buffered Milk Consumption Protocol**

In this test, the milk solution will be buffered with baking soda to determine if milk consumption can be reduced.

The same milk relative hydraulic conductivity and consumption protocol will be followed except baking soda will be added to the milk solution. Proportionally add enough baking soda to 100 mLs of non-fat milk to neutralize the stored acidity in the waste rock sample as determined in the beaker test screening. Follow the same protocol and compare the results

#### **Equipment & Materials**

Same as non-buffer milk

Baking soda (100 grams or as needed)

#### **Whey/Lactose Consumption Protocol**

<u>Background</u> - This technology might be applied in the Saturated Zone of the Waste Rock. Lactose/whey would support microbial fermentation to organic acids such as lactic acid (homolactic fermentation) or other compounds in heterolactic fermentations (lactic acid, acetic acid, ethanol, hydrogen etc.) with manure inoculum containing fermenting bacteria and sulfate reducing bacteria. Lactic acid (lactate) is frequently the primary carbon source added to growth media for many types of sulfate reducing bacteria, with hydrogen, ethanol or sometimes even acetate potentially supporting growth of various members of this physiological group (see Bergey's Manual of Determinative Bacteriology) - this is also why acidic sour milk works best instead of fresh milk, as per WRI technology.

The presence of high concentrations of lactate (with micronized limestone to buffer microenvironment) could allow rapid growth of anaerobic sulfate reducers, producing additional alkalinity and H<sub>2</sub>S to precipitate metals. In other words, the initial subsurface SRB activity and alkalinity/H<sub>2</sub>S production would be jump started. And less reactive, longer lived carbons sources could also be added without interfering with this early phase activity.

The acidity in the pit lake and the waste rock pore water will dehydrate lactose or other sugars to elemental carbon. While lactose may be more resistant to dehydration losses compared to sucrose, this effect needs to be assessed so that add enough lactose carbon is added to support microbial activity and whether adding buffering alkalinity would be helpful. It is uncertain if this reaction occurs in a reasonable time scale at low temperatures and with acidic MIW. Lactose is assayed after boiling in hydrochloric acid – the bond linking two sugar monomers is hydrolyzed, but the sugar monomers are present. To validate the procedure, the team will run water activity analyses for a few transition or unsaturated zone samples with a Decagon Pawkit water activity meter to determine if contact with the solids and amendments affects this to the point where microbial activity might be negatively impacted.

#### Whey Relative Hydraulic Conductivity and "Consumption" Tests

Follow the protocol for the milk solution without baking soda to determine relative hydraulic conductivity and consumption. Run just one 100ml of solution through the solids as whey will not have the same plugging properties as milk. If alkalinity adjustments are warranted another test can be performed following the buffered milk protocol. Again just 100ml of solution would be run through.

#### **Equipment & Materials**

Same as non-buffer and buffered milk tests

#### Whey Consumption Due to Hydration Test

One-liter samples of pit water and ground water will be titrated with granulated sugar (as a baseline) and powdered lactose as separate tests. Sugar content in the solutions will be measured using a hydrometer. See https://www.youtube.com/watch?v=oj77Gf0T5SE

One-liter containers of tap water will be titrated with sugar and lactose and the hydrometer as a control. Adjustments to hydrometer readings for lactose content will be assessed.

After the effects of sugar/lactose dehydration are determined, the remaining sugar/lactose in solution would be available for microbial activity.

#### Equipment & Materials

- 1 liter pit water
- 1 liter ground water
- 2 liters tap water
- Balance (1kg cap.)
- Granulated sugar 500 grams

- Powdered whey/lactose (500 grams)
- Sugar content hydrometer
- Misc. beakers
- Decagon Pawkit water activity meter

#### **Barite Hill Source Control Technology Descriptions**

#### Sodium Lauryl Sulfate (SLS)

The iron-oxidizing bacteria, Acidithiobacillus ferrooxidans (ATBFO), have proven to play a critical role in creating acid rock drainage (ARD) and are strongly associated with the formation of acid mine drainage (Kleinmann, et.al. 1981; Schrenk et. al., 1998). Metals found in mine soils, including iron, manganese, aluminum, magnesium, lead, copper, zinc, cadmium, and selenium are solubilized, creating drainage that is toxic to the environment. Suppression of ATBFO has proven to significantly reduce the generation of ARD at mine sites (Rastogi 1996).

Anionic surfactants are effective inhibitors of *ATBFO*, as they destroy the integrity of the cytoplasmic membrane of the bacteria thus allowing the acid that they create to enter the cells and destroy them.

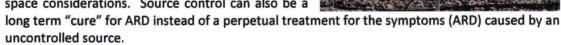
As of the mid-1990's, anionic surfactants had only been applied to surface sources of ARD. That changed when Plocus and Rastogi (1997) demonstrated that subsurface application of anionic surfactants using injection techniques could successfully reduce ARD at the Fisher Site in Banks Township, Indiana County, Pennsylvania. This was accomplished by identifying the acid-generating zones using geophysical techniques and designing a multiple-stage borehole based injection program that targeted the ARD "hot spots".

Geophysical mapping with electromagnetic terrain conductivity meters and magnetometers were utilized to identify pyritic zones which were responsible for the high acid production on the Fisher Site.

SLS could be used in conjunction with waste milk (described below) to develop long term ARD source control. It might be applied with inexpensive drip irrigation technology used in mining operations.

#### Additional issues to be considered:

 Source control is always preferable to active or passive treatment because there is no permanent constructed infrastructure, and consequently no space considerations. Source control can also be a



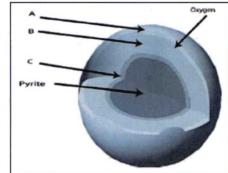
- Application rates, doses, and methods need to be tested so that adequate penetration can be achieved without significant surface run off.
- When used in conjunction with waste milk and a strong vegetation cover, long-term acid generation reduction can be achieved.
- Cost is very low with virtually no maintenance costs.
- This source control technique would be most effectively applied to the unsaturated and transition zones of the waste rock dump.
- For the effects of this source control option to become permanent, it needs to be implemented
  with a robust vegetative cover which would be installed anyway to minimize erosion and water
  contact with the mine waste. This situation already exists at Barite Hill.
- Surficial drip irrigation application on the waste rock dump surface is impractical due to the
  existence of the cover. Subsurface injection or delivery techniques would be required.



#### Milk and Whey

Researchers at the Wyoming Research Institute (Jin 2008) determined that a biofilm nurtured by waste milk or other dairy products, inoculated with a "probiotic" bacterial community, could out-compete Acidithiobacillus ferrooxidans (ATBFO) on the surfaces of pyrite grains and thus suppress acid mine drainage (ARD).

In essence, the milk produces a biological environment unfavorable to acid-forming bacteria until a vegetative cover can be established to perpetuate the favorable environment. It can also be used in a saturated environment. When ATBFO are present, waste milk is best used in conjunction with Sodium Lauryl Sulfate (SLS) to develop long term source control; milk would probably be applied in a diluted solution (strength TBD) or in powder form that would be delivered to pyritic mine waste during a rainy interval.



The concept of introducing competing bacteria is not new. Sobek, Benedetti, and Rastogi (1990) suggested that a probiotic process would complement the application of a slow-release acidophilic bactericide, sodium lauryl sulfate:

"Inhibiting or destroying thiobacilli can significantly slow the rate of acid production. Anionic surfactants, organic acids and food preservatives (Onysko et al. 1984) act as bactericides and kill these bacteria; however; bactericides degrade over time and are lost because of leaching and runoff. To overcome the inherent short duration effectiveness of spray applications, controlled release systems to provide the bactericide slowly over a long time period were developed (Sobek et al. 1985).

Control of acid generation for prolonged periods greatly enhances reclamation efforts and can reduce reclamation costs by reducing the amount of topsoil needed to establish vegetation. Three natural processes resulting from strong vegetative cover for three years or more can break the acid production cycle. These processes are:

- 1) A healthy root system that competes for both oxygen and moisture with acidproducing bacteria;
- 2) Populations of beneficial heterotrophic soil bacteria and fungi that are reestablished, resulting in the formation of organic acids that are inhibitory to *T. ferrooxidans* (Tuttle et al. 1977); and
- 3) The action of plant root respiration and heterotrophic bacteria increase CO<sub>2</sub> levels in the spoil, resulting in an unfavorable microenvironment for growth of *T. ferrooxidans.*"

Sobek, Benedetti, and Rastogi viewed antibacterial application as a method to reduce the volume of topsoil needed to revegetate potentially acid generating or PAG waste. They believed that at least three years of acidophilic bacterial suppression was sufficient to accomplish this goal.

Additional issues to be considered:

- Source control is always preferable to active or passive treatment because there is no permanent constructed infrastructure, and consequently no space considerations.
- Application rates, doses, and methods (subsurface injection, infiltration, or horizontal migration), would need to be tested so that adequate penetration can be achieved.

- As with SLS, for the effects of this source control option to become permanent, it needs to be implemented with a robust vegetative cover as subsequently discussed. The root zone of the vegetative cover provides a sustainable supply of organic acids which produce the same effect as the waste milk for the long term.
- When used in conjunction with SLS, a long-term acid generation reduction can be achieved. Prevention is more desirable than perpetual treatment.
- Cost is very low with virtually no maintenance costs.
- Typical treatment process includes applying SLS first, then waste milk, depending on the situation. For example, SLS would not be used in a saturated zone because pyrite oxidation in that environment is abiotic, driven by the presence of ferric iron.
- As with the SLS technology, the mine waste must exhibit reasonable permeability to allow
  adequate penetration to be considered practical. Preliminary screening test results might show
  that the mine wastes may not be permeable and that the congealing of milk when it contacts the
  acidic material makes a nominal plugging situation worse.

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